Investigation of high-temperature platinum resistance thermometers at temperatures up to 962 °C, and, in some cases, 1064 °C

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ABSTRACT

The stability of high temperature standard platinum resistance thermometers, having nominal resistances of 0.25 to 2.5 ohms, and their use in the realization of the International Temperature Scale of 1990 over the range of 0 °C to 961 °C were investigated. Special procedures were employed for their use above 500 °C. These techniques involved annealing and protection against contamination. A sodium heat-pipe furnace was used to realize the freezing points of aluminum, silver, and gold. The results to be presented yielded information on non-uniqueness, subrange inconsistencies and the effects of heating above 600 °C.

SUBJECT INDEX: International Temperature Scale of 1990 (ITS-90), Platinum resistance thermometers and Thermometry, High temperature thermometry

INTRODUCTION

For the International Temperature Scale of 1990 (ITS-90) (1), the range of standard platinum resistance thermometers (SPRTs) was extended upward to the freezing-point temperature of silver (961.78 °C). This reduced the uncertainty of temperature measurements that resulted from use of type S thermocouples of the International Practical Temperature Scale of 1968, amended edition of 1975 (IPTS-68) (2), over the range 630 °C to 1064 °C. Specially constructed, low-resistance SPRTs are used for determining temperatures above the freezing point of aluminum (660.323 °C) (3); these are known as high-temperature SPRTs (HTSPRTs). Their use for defining the ITS-90 amplifies the importance in understanding their capabilities. Repeated calibrations and exposure of HTSPRTs to high temperatures yield results on their stability (4), reproducibility, non-uniqueness and subrange inconsistencies. Results of such an investigation conducted with several types of HTSPRTs will be presented.

EXPERIMENTAL DETAILS

The types of HTSPRTs used in this study had nominal resistances at the triple point of water (TPW) that ranged from 0.25 Ω to 2.5 Ω, as shown in Table I. The 0.59 Ω HTSPRTs were constructed at the D. I. Mendeleyev Institute of Metrology (VNIIM); they consisted of a dual, coiled-helix platinum winding on a twisted blade of fused silica. This construction of the element allows the coiled helix of platinum wire to expand freely along its length. The resistance elements of the other thermometers investigated used other well-known designs.

Eighteen of the HTSPRTs studied were constructed at VNIIM. Of those, I and J will be referred to as NIST/VNIIM HTSPRTs; they were investigated at NIST. The other 16 (K-Z) were investigated at VNIIM. The remaining eight (A-H) HTSPRTs were obtained from three commercial sources and their nominal resistance values were 0.25 Ω or 2.5 Ω. The thermometers from commercial-sources were used only up to the silver freezing point but the two NIST/VNIIM HTSPRTs (I and J) were calibrated up to the freezing point of gold before and after being subjected to temperatures above 962 °C for extended periods of time.

HTSPRTs (K-Z) were calibrated at VNIIM up to the freezing point of gold. After calibration at the aluminum, silver and gold freezing points, these thermometers were annealed in a vertical furnace at 650 °C for 3 hours and then measured at the triple point of water. All thermometer measurements at VNIIM were made with a Guildline model 9975 bridge[a].

The automated measurement system for calibration of HTSPRTs at NIST includes a commercially-available, automatic, ac resistance-ratio bridge, operating at a frequency of 30 Hz. Different valued reference resistors were used with this bridge in order to increase the resolution and to minimize measurement error from non-linearity. The data were taken automatically via a computer-controlled IEEE-488 bus and logged to a data file for later analysis. All bridge measurements of the HTSPRTs were conducted at two excitation currents to permit analysis of the results at zero-power dissipation. A 1-Ω reference resistor and excitation currents of 10 mA and 14.14 mA were used in making measurements on the 0.25-Ω thermometers; a 10-Ω reference resistor and excitation currents of 7.07 mA and 10 mA for the 0.59-Ω thermometers; and a 10-Ω reference resistor and excitation currents of 5.0 mA and 7.07 mA for the 2.5-Ω thermometers. The resolution of measurements using the commercial bridge and the reference resistors indicated was the equivalent of 0.01 m°C for the 2.5 Ω and 0.25 Ω HTSPRTs and 0.04 m°C for the 0.59 Ω HTSPRTs.

Table I. List of HTSPRTs investigated.

HTSPRT	Type, Ω	Coil Description	Coil Support
A	0.25	bird-cage	bird cage
B	0.25	single-layer bifilar	notched blade
C	0.25	single-layer bifilar	notched cross
D	0.25	single-layer bifilar	notched blade
E	2.5	coiled helix	notched cross
F	0.25	single-layer bifilar	notched blade
G	0.25	bird-cage	bird cage
H	2.5	coiled helix	notched cross
I	0.59	dual coiled helix	twisted blade
J	0.59	dual coiled helix	twisted blade
K	0.59	dual coiled helix	twisted blade
L	0.59	dual coiled helix	twisted blade
M	0.59	dual coiled helix	twisted blade
N	0.59	dual coiled helix	twisted blade
O	0.59	dual coiled helix	twisted blade
P	0.59	dual coiled helix	twisted blade
Q	0.59	dual coiled helix	twisted blade
R	0.59	dual coiled helix	twisted blade
S	0.59	dual coiled helix	twisted blade
T	0.59	dual coiled helix	twisted blade
U	0.59	dual coiled helix	twisted blade
V	0.59	dual coiled helix	twisted blade
W	0.59	dual coiled helix	twisted blade
X	0.59	dual coiled helix	twisted blade
Y	0.59	dual coiled helix	twisted blade
Z	0.59	dual coiled helix	twisted blade

After a thermometer was properly annealed (5), it was calibrated at the following fixed points, in the sequence listed: TPW (0.01 °C), Au (1064.18 °C), TPW, Ag (961.78 °C), TPW, Al (660.323 °C), TPW, Zn (419.527 °C), TPW, Cd (321.069 °C), TPW, Sn (231.928 °C), TPW, In (156.5985 °C), TPW, Ga (29.7646 °C) and TPW. Only the VNIIM-made thermometers, however, were exposed to temperatures of the Au freezing point. Calibration at "redundant fixed points" (Cd, In and Ga) allow analysis of the scale non-uniqueness at those points. The multiple combinations of fixed points yield values of subrange inconsistencies.

The realization of the Au freezing point was accomplished by heating the ingot of metal overnight to about 5 °C above the freezing point, then inserting a heated HTSPRT into the cell, waiting for equilibrium, then reducing the furnace temperature to a value 1 °C below the Au freezing point. The supercool was monitored with the HTSPRT until recalescence was observed. Then, the thermometer was removed to the auxiliary furnace (at 1072 °C) and a clean fused-silica rod, initially at room-temperature, was repeatedly inserted into and removed from the reentrant well at one minute intervals over a three-minute period. The HTSPRT was then re-inserted into the fixed-point cell and, after a 1-hour wait for thermal equilibration, measurements were conducted. The construction of the gold freezing-point cell is similar to that of the silver freezing-point cell, which is described in another paper at this Symposium (5).

When used at temperatures above 500 °C, HTSPRTs require special handling to maximize their accuracy in temperature measurements and to minimize their degradation. Some of the precautions that were taken were as follows. First, care was taken to protect the fused-silica sheath of the thermometer from devitrification. Devitrification, the process in which the fused-silica crystallizes, may result from the presence of any oils or salts that are transferred to the sheath from the user's hands as the thermometer is placed into an auxiliary furnace or into a fixed-point reentrant well. The devitrification is irreversible and will cause the sheath to become very brittle. Consequently, in order to prevent this from happening, the sheath of the SPRT was cleaned thoroughly with an isopropanol-soaked, clean chemical wipe. Furthermore, when touching the fused-silica sheath, polyethylene gloves were worn to prevent re-contamination. It is important to remember that contaminants of salt and oil on the sheath of a thermometer which is placed in a fixed-point cell may be transferred to the fused-silica reentrant well of that cell.

Second, the resistance element of the thermometer was protected from contamination by metal ions diffusing through the fused-silica sheath and alloying with the platinum at high temperatures. The metals that contribute to the degradation of HTSPRTs are considered to be primarily nickel and copper. Consequently, thermometers that are placed in an auxiliary furnace to be heated before insertion into a fixed-point cell or to be cooled to 500 °C will be subject to contamination from any inconel and or nichrome used in construction of the furnace. Hence, for protection of HTSPRTs when they are in auxiliary furnaces, NIST uses a system that consists of a platinum test tube, with a wall thickness of 0.13 mm and 66 cm long, that is located between two 66 cm long fused-silica test tubes. The fused-silica tubes protect the platinum tube from physical damage. This reference-grade (99.9% pure) platinum sheath acts as an impurity acceptor, thus protecting the HTSPRT from degradation. Thermometers that are in fixed-point cells would not be contaminated since they are surrounded by high-purity graphite enclosing high-purity metals (Al, Ag or Au).

A third precaution taken was to minimize thermal shock to the HTSPRT and the quenching in of lattice-site defects. Allowing the thermometer to cool quickly by removing it from a furnace at 962 °C to ambient conditions will cause the resistance at 0.01 °C to increase. This increase in resistance will cause an error in $W(t_{90})$ [$W(t_{90})=R(t_{90})/R(0.01\ °C)$]. The annealing procedure, using auxiliary furnaces, for HTSPRTs used up to the freezing point of silver was as follows: heat the HTSPRT from 500 °C to 970 °C over a period of 2 hours, keep at 970 °C for 0.5 hours, cool from 970 °C to 500 °C over a period of 4 hours, then remove the thermometer to ambient conditions. Thermometers used up to the freezing point of gold were annealed at 1072 °C for 0.5 hours and then cooled to 500 °C over a period 5 hours. These periods of time for cooling the HTSPRTs were selected after conducting tests of successively longer cooling times, ranging from 1 hour to 8 hours, to determine the optimum time for cooling the HTSPRT to minimize deleterious effects on the resistance at the triple point of water, but yet not be unduly long.

RESULTS AND DISCUSSION

HTSPRTs that are used as standards in defining the ITS-90 over the range from 0 °C to 961.78 °C must meet the criteria: $W(29.7646\ °C) \geq 1.118\ 07$ and $W(961.78\ °C) \geq 4.284\ 4$. The values of $W(29.7646\ °C)$ and $W(961.78\ °C)$ for the HTSPRTs investigated are given in Table II. For thermometers A through H of Table I, the average value for $W(29.7646\ °C)$ is 1.118 145 458 and for $W(961.78\ °C)$, the average is 4.286 445 129. The average values for HTSPRTs I and J, each with four calibrations, are 1.118 118 889 for $W(29.7646\ °C)$ and 4.285 925 033 for $W(961.78\ °C)$. The average value for VNIIM-calibrated thermometers K through Z is 4.285 750 0 for $W(961.78\ °C)$, with no data available for $W(29.7646\ °C)$. Thus, the 10 NIST-calibrated HTSPRTs and the 16 VNIIM-calibrated HTSPRTs of Table I all meet the ITS-90 criteria for defining standards of the scale. Additionally, although not a criterion of the ITS-90, the average value for $W(1064.18\ °C)$ is 4.571 328 621 for thermometers I and J and 4.570 780 3 for thermometers K through Z. The results on this set of HTSPRTs, show that the assigned criteria for use up to the freezing point of silver are not very stringent.

Table II. $W(t_{90})$ values at the gallium melting point, the silver freezing point and the gold freezing point, and the equivalent temperature change at the TPW during calibration. The designations such as I1 and J3, refer to the calibration number of HTSPRT I and J, respectively.

HTSPRT	W(Ga)	W(Ag)	W(Au)	Change in TPW, m°C
A	1.118127759	4.284973039		1.8
B	1.118153372	4.286681759		1.0
C	1.118137099	4.286294350		0.3
D	1.118140398	4.286402239		0.4
E	1.118147846	4.287004478		0.6
F	1.118156491	4.286859919		0.7
G	1.118155120	4.286833706		0.7
H	1.118145579	4.286511539		0.8
I1	1.118131026	4.286264175	4.571665167	1.0
I2	1.118132274	4.286241902	4.571631458	0.8
I3	1.118130758	4.286223437	4.571603868	1.2
I4	1.118129094	4.286189515	4.571559761	0.7
J1	1.118107935	4.285640208		0.6
J2	1.118111806	4.285648574	4.570993361	0.3
J3	1.118106108	4.285618700	4.570950063	0.2
J4	1.118102108	4.285573755	4.570896665	0.7
K		4.2853441	4.5703994	
L		4.2858254	4.5709062	
M		4.2858480	4.5708915	
N		4.2859215	4.5710263	
O		4.2858899	4.5709621	
P		4.2855115	4.5704879	
Q		4.2856634	4.5706507	
R		4.2855490	4.5706130	
S		4.2854060	4.5702502	
T		4.2857728	4.5708371	
U		4.2854824	4.5704275	
V		4.2858152	4.5709498	
W		4.2856877	4.5707044	
X		4.2866338	4.5716249	
Y		4.2858771	4.5709164	
Z		4.2857728	4.5708370	

One method of determining the stability of HTSPRTs is measurement of the resistance value of the HTSPRT at the TPW following the measurement at every other temperature, such as that of a freezing-point cell. During calibration of an HTSPRT at temperatures up to the freezing point of silver, following the measurement sequence stated above, the thermometer is read eight times at the TPW. NIST has set as acceptable a change in resistance equivalent to 2.0 m°C at 0.01 °C during the entire calibration sequence. During calibrations through the freezing point of silver, HTSPRTs A through H showed equivalent changes at zero-power dissipation as indicated in Table II. None had a change as large as 2 m°C and their average change at the TPW was equivalent to 0.8 m°C. Thermometers I and J, which were calibrated at fixed points through the freezing point of gold, showed the equivalent changes at zero-power dissipation as indicated also in Table II. Their average change was equivalent to 0.7 m°C.

An extension of the method given above for determining the stability of an HTSPRT is measurement of the resistance value of the HTSPRT at the TPW following exposure of the thermometer for long times to high temperatures. HTSPRTs I and J experienced such long term exposures between each calibration. They were used in a thermocouple *emf* versus t_{90} study (6,7,8,9) and in that investigation they were subjected to elevated temperatures for long times. Calibration of these thermometers after long-term exposure to temperatures up to 1070 °C for HTSPRT I and 1002 °C for HTSPRT J yielded results on their stability with time of exposure to the high temperatures. In each calibration, the HTSPRTs were calibrated from 0.01 °C to the freezing point of gold. Table III shows the heating times at temperatures above 500 °C that each of these HTSPRTs received between calibrations.

Table III. Time in hours that HTSPRTs were held above given temperatures, and equivalent changes at TPW after the heat treatments.

HTSPRT I			
	Duration of heat treatment given to HTSPRT between designated calibrations		
t_{90}, °C	I1 to I2	I2 to I3	I3 to I4
500	675	300	610
600	530	210	420
700	325	120	325
800	240	80	270
900	180	50	151
1000	110	20	20
1050	47		
max t_{90}	1070	1004	1001
Change at TPW, m°C	3.5	3.9	4.5

HTSPRT J			
	Duration of heat treatment given to HTSPRT between designated calibrations		
t_{90}, °C	J1 to J2	J2 to J3	J3 to J4
500	26	180	405
600	21	110	250
700	15	85	155
800	12	45	110
900	10	25	55
1000		15	20
max t_{90}	970	1001	1002
Change at TPW, m°C	-3.3	4.6	4.5

Table III also shows the effects of the heat treatments and calibrations, with routine annealing, on the resistance of these thermometers at the TPW. The resistance of thermometer I at the TPW increased by the equivalent of 3.5 m°C after the first heat treatment of 530 hours above 600 °C, an additional 3.9 m°C after a second heating of 210 hours above 600 °C and an additional 4.5 m°C after a third heat treatment of 420 hours above 600 °C. Thermometer J decreased in resistance at the TPW by the equivalent of -3.3 m°C, after minimal heat treatment of 21 hours above 600 °C. This was followed by an increase of 4.6 m°C after a second heating of 110 hours above 600 °C, and an additional 4.5 m°C after a third heat treatment of 250 hours above 600 °C. During these heat treatments, the resistances of HTSPRTs I and J were measured at the TPW during four separate calibrations to the silver freezing point and they had equivalent maximum temperature changes of 12 m°C; W(Ag) had maximum equivalent changes of 27 m°C.

Figures 1 and 2 show the effect of exposure to high temperatures on the reproducibility of the calibration for the two NIST/VNIIM thermometers. The differences are relative to the previous calibration for each thermometer, i.e., after a routine anneal. The changes in the three successive calibrations of HTSPRT I showed a continuous decrease in the $W(t_{90})$ values, with an increase in the resistance at 0.01 °C. Thermometer J received two successive calibrations with minimal time at elevated temperatures (see Table III) and did not change by more than the equivalent of 3 m°C at the silver freezing point. Heating of this thermometer at temperatures up to 1002 °C, however, caused a larger change in the measured $W(t_{90})$ at the fixed points.

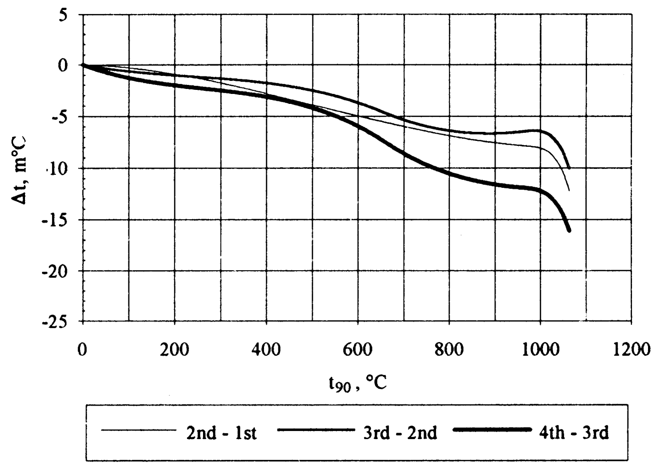

Figure 1. Curves for HTSPRT I showing the effect that heat treatment to a minimum of 1000 °C has on the reproducibility of the calibration of the thermometer. The differences are relative to the previous calibration.

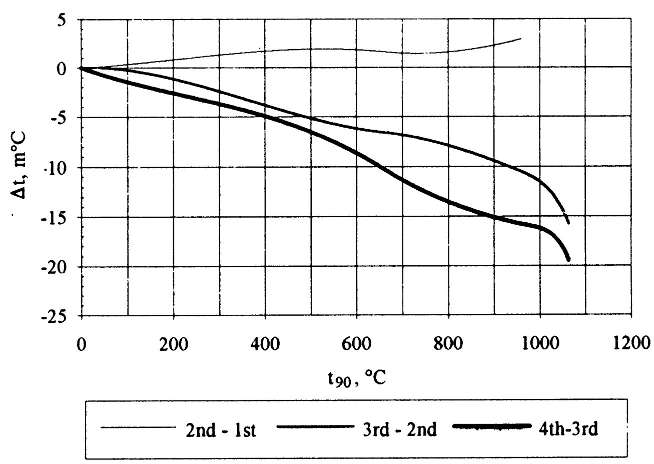

Figure 2. Curves for HTSPRT J showing the effect that heat treatment to a minimum of 1000 °C has on the reproducibility of the calibration of the thermometer. The differences are relative to the previous calibration.

Figure 3 shows the equivalent temperature drift in the TPW resistance of thermometer I (solid triangles) and thermometer J (open diamonds). These thermometers were subjected to an initial annealing at 1090 °C for 60 hours. The heat treatment data from 0 to 115 hours were obtained at VNIIM, and at NIST for times greater than 115 hours. The resistance of HTSPRT I at the TPW drifted approximately 3 m°C per 100 hours of heat treatment above 1000 °C, whereas that for HTSPRT J drifted approximately 10 m°C per 100 hours above 1000 °C.

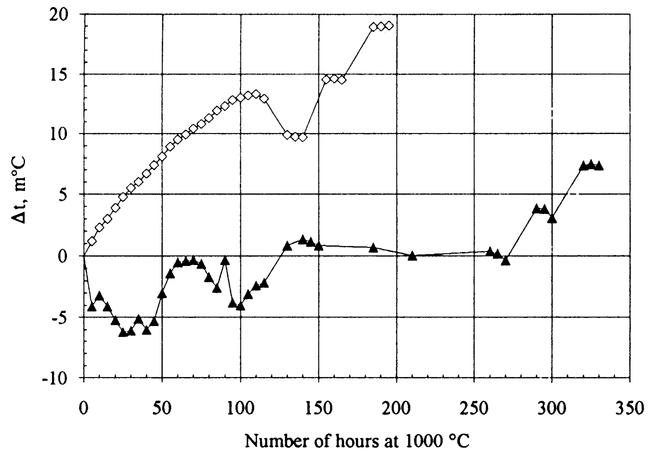

Figure 3. Equivalent temperature drifts of the two NIST/VNIIM HTSPRTs at the TPW after heat treatment above 1000 °C. Results for the first 115 hours for each thermometer reflect measurements made at VNIIM. The open diamonds represent HTSPRT J and the solid triangles represent HTSPRT I.

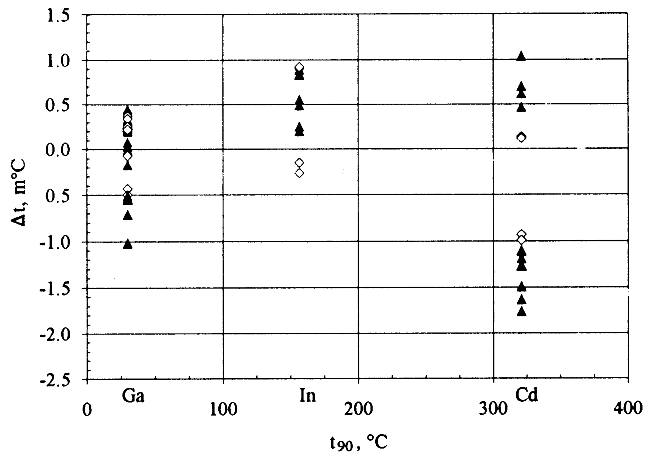

Figure 4. Non-uniqueness at the triple point of gallium, freezing point of indium and the freezing point of cadmium for all possible temperature subranges for eight HTSPRTs (A-H). The open diamonds represent the 2.5 Ω HTSPRTs and the solid triangles represent 0.25 Ω thermometers.

Other NIST HTSPRTs, repeatedly measured at the freezing point of silver and the triple point of water, have experienced less of a change at the TPW. The change at the TPW for the three NIST Ag-point check thermometers (0.25 Ω and 2.5 Ω) ranged from -0.7 m°C to 1.8 m°C during 28 calibrations to the silver freezing point and the average drift is approximately 0.7 m°C per 100 hours at the silver point. During this time, the resistance at the silver freezing point changed by the equivalent of 1.7 m°C (maximum for the three HTSPRTs) and the W(Ag) changed by the equivalent of 2.3 m°C (maximum for the three HTSPRTs).

Additional data from VNIIM give results on equivalent temperature changes for HTSPRTs at the TPW after repeated measurements (minimum of three) at the Au, Ag, Al, Zn and Sn freezing points. One of those HTSPRTs had a maximum change of 12 m°C. The others had maximum changes that ranged from 2 m°C to 6 m°C, with an average change of 5 m°C.

Calibration of HTSPRTs over the temperature range 0 °C to either 961.78 °C or 1064.18 °C allows for measurement of the thermometer at "redundant" fixed points not used in deriving the deviation function. These redundant fixed points may be used in a determination of the non-uniqueness of the ITS-90 at those temperatures. In general, different thermometers exhibit slight differences in indicated temperature at any given point intermediate to those of the defining fixed points used in calibration. This non-uniqueness is determined by the temperature difference between the $W(t_{90})$ calculated from the deviation function (determined by calibration) and the $W(t_{90})$ measured at an intermediate defining fixed-point temperature. The Cd, In, and Ga fixed points are "redundant" for several of the temperature subranges, as defined by the ITS-90. Using all possible temperature subranges, an estimate of the non-uniqueness may be determined at the freezing point of Cd using two subranges, at the freezing point of In using two subranges, and at the triple point of Ga using four subranges.

Calibration of a large number of HTSPRTs makes it possible to determine a preliminary value for the non-uniqueness of the ITS-90 at these "redundant" fixed points. Results for eight (A-H) HTSPRTs investigated at the redundant fixed points for all of the possible temperature subranges are shown in fig. 4. The temperature spread for the 2.5 Ω HTSPRTs (open diamonds) is ±0.6 m°C at the Cd freezing point, ±0.6 m°C at the In freezing point and ±0.4 m°C at the Ga triple point. The temperature spread for the 0.25 Ω HTSPRTs (closed triangles) is ±1.4 m°C at the Cd freezing point, ±0.3 m°C at the In freezing point and ±0.7 m°C at the Ga triple point. Figure 5 shows the non-uniqueness as a function of time and elevated heat treatment for four calibrations of the NIST/VNIIM HTSPRTs. The temperature spread for HTSPRTs I and J is ±1.6 m°C at the Cd freezing point, ±0.5 m°C at the In freezing point and ±0.4 m°C at the Ga triple point. There is no apparent trend in the non-uniqueness values of HTSPRTs I and J for the four calibrations, although the values at the Cd freezing point are larger than would have been expected.

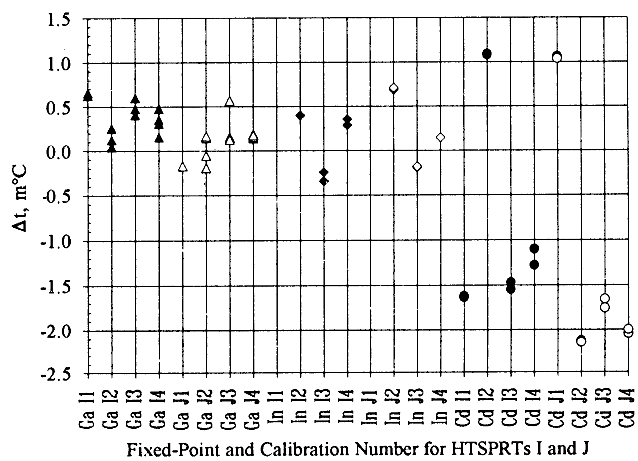

Figure 5. Non-uniqueness at the triple point of gallium, freezing point of indium and the freezing point of cadmium for all possible temperature subranges for the two NIST/VNIIM HTSPRTs. The solid symbols represent data for HTSPRT I and the open symbols represent data for HTSPRT J.

The overall spread of non-uniqueness for the complete set of HTSPRTs (A-J) in this study is ±1.6 m°C at the Cd freezing point, ±0.6 m°C at the In freezing point and ±0.6 m°C at the Ga triple point.

The flexibility of the ITS-90 allows thermometers to be calibrated over different temperature subranges. These subranges overlap in specific regions, but use different sets of defining fixed points for calibration and, consequently, involve different deviation functions. These different deviation functions for a given thermometer yield different calculated values of temperature for a measured $W(t_{90})$. This phenomenon is known as subrange inconsistency.

The subrange inconsistencies for HTSPRTs are of interest for only the subrange 0 °C to 419.527 °C, relative to the subrange 0 °C to 660.323 °C. The HTSPRT is not recommended for use with lower temperature subranges. Figure 6 shows the subrange inconsistencies for the eight 0.25 Ω and 2.5 Ω thermometers. The results for the 2.5 Ω HTSPRTs are indicated by the thick-lined curves. The maximum deviation for the 2.5 Ω thermometers is about 0.4 m°C and the total spread is about 0.7 m°C. The maximum deviation of all the curves is about 0.7 m°C and the total spread is about 1.2 m°C. Figure 7 shows the subrange inconsistencies for NIST/VNIIM HTSPRTs I and J. The curves for thermometer J are designated by thick lines and those for thermometer I are designated by thin lines. The four different lines for each thermometer represent results from four different calibrations. The maximum deviation is about 0.2 m°C and the total spread is about 0.3 m°C. Figure 8 shows the subrange inconsistencies for 14 VNIIM-calibrated HTSPRTs K through X. The maximum deviation is about 2.9 m°C and the total spread is about 4.8 m°C with HTSPRTs O, T and V being the three outliers. Exclusion of these outliers gives a maximum deviation of about 0.4 m°C and a total spread of about 0.7 m°C.

The ITS-90 stipulates that the platinum resistance thermometer is the defining interpolation instrument from 13.8033 K to 961.78 °C. Investigations of *emf* versus t_{90} relationships for types S, R and B, and Au/Pt thermocouples (6,7,8,9) by comparison with an HTSPRT (I or J) up to 1070 °C, however, led to the need for determining temperatures above 962 °C with those HTSPRTs. Various mathematical models of $W(t_{90})$ as a function of t_{90} were investigated for use in interpolating over the undefined (in terms of HTSPRTs) temperature range between the silver and the gold freezing-point temperatures. The ITS-90 reference function for use above 0 °C was derived by fitting $W(t)$ data that extended up to 989 °C (10). The extrapolation of the reference and deviation functions of the ITS-90 up to the freezing point of gold gives an error at the gold freezing point of about 100 m°C for the NIST/VNIIM HTSPRTs I and J.

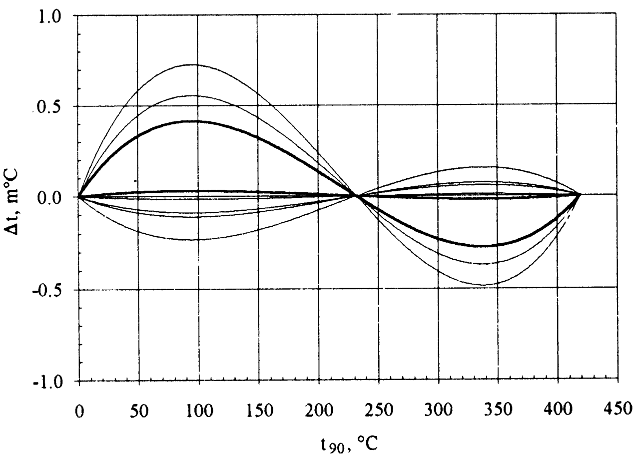

Figure 6. The curves show the subrange inconsistencies for the subrange 0 °C to 419.527 °C relative to the subrange 0 °C to 660.323 °C for eight HTSPRTs (A-H). The 0.25 Ω HTSPRTs are indicated by the seven thin lines and the 2.5 Ω HTSPRTs are indicated by the two thick lines.

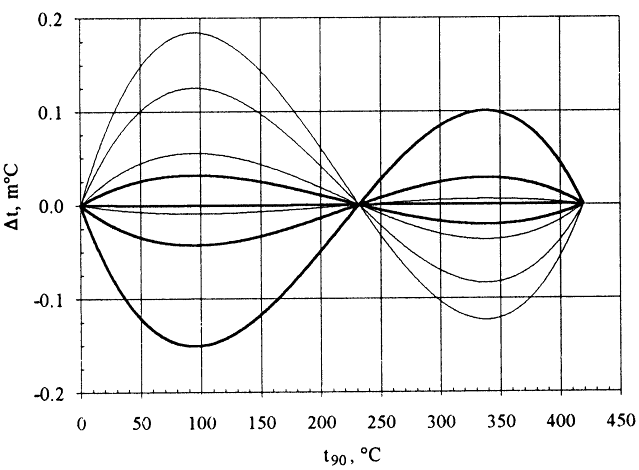

Figure 7. The curves show the subrange inconsistencies for the subrange 0 °C to 419.527 °C relative to the subrange 0 °C to 660.323 °C for two NIST/VNIIM HTSPRTs. The inconsistencies from the four calibrations of thermometer I are indicated by the thin lines and the inconsistencies from the four calibrations of thermometer J are indicated by the thick lines. One of the thick lines is indistinguishable from the grid line at 0.0 m°C.

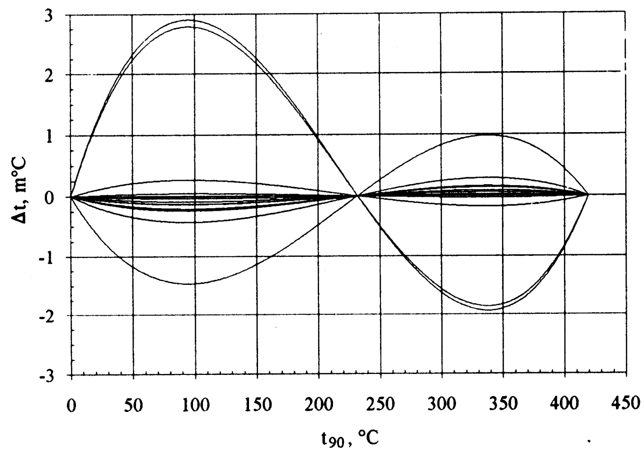

Figure 8. The curves show the subrange inconsistencies for the subrange 0 °C to 419.527 °C relative to the subrange 0 °C to 660.323 °C for 14 of the VNIIM HTSPRTs (K-X).

The first model of extrapolation that we attempted was to add an additional term, $e[W(t_{90})-W(Ag)]^2$, to the prescribed deviation function used up to 961.78 °C. Here, e = 0 at temperatures below 961.78 °C. Certainly, extrapolation of the reference function beyond its intended range leads to error and using this model will lead to considerable uncertainty in temperature values; however, this model was used to establish a baseline for our other models.

From fitting $W(t_{90})$ vs. t_{90} data obtained from the ITS-90 high-temperature reference function (and using the original data (10) up to 989 °C), we determined that a 4th degree polynomial described the data quite well over the range from about 850 °C to 970 °C, with the residual error not exceeding ±1 m°C . The use of higher order polynomials did not improve the fit. Based on these results, it seemed reasonable that a similar 4th degree polynomial could be used for determining temperatures from 961 °C to 1070 °C. Using data at a variety of temperatures, four different fits of $W(t_{90})$ vs. t_{90} data were generated from the results of a calibration of the HTSPRT through the freezing point of gold. The maximum disagreement among the four fits did not exceed 8 m°C. The 4th degree polynomial chosen for use had as the argument $[(t_{90}-995)/75]$ and was based on fits to $W(t_{90})$ data at 925 °C, 940 °C, 955 °C, 961.7807 °C [Ag plus hydrostatic head (HH)], and 1064.1813 °C (Au+HH) and the close matching of the slope of the function with that of the ITS-90 deviation function at the freezing point of silver. For the various fits, differences from the baseline did not exceed 13 m°C. The differences in the slopes of $W(t_{90})$ at the freezing point of silver between the ITS-90 deviation function and the 4th degree polynomials based on a given calibration did not exceed 1.2E-07/°C for any of the four fits. For the polynomial chosen to be used, the difference did not exceed 2.2E-08/°C for four calibrations of HTSPRT I and three calibrations of HTSPRT J. The 4th degree polynomial selected was used for determining temperatures above the silver freezing point in the comparison experiments on thermocouples (6,7,8,9). The estimated error in the determination of temperature using that polynomial is not expected to exceed ±10 m°C, with the maximum deviation occurring at 1030 °C.

The uncertainties associated with a calibration of an HTSPRT are based on the stability and reproducibility of the defining fixed points, the annealing heat treatment and stability of that thermometer, and the uncertainty of the measurement equipment. An assessment of total uncertainty for an HTSPRT calibration is important in the determination of the user's total uncertainty in their use of the thermometer. The estimated total calibration error for each temperature subrange is determined from the propagated errors from all of the defining fixed points involved. The error propagated from each defining fixed point is calculated for each subrange by propagating the error associated with that fixed point, using the appropriate deviation function, and assuming that there is no error at the other fixed points (3). The total error for a given t_{90} is determined from the root sum square (RSS) error arising from the various components from all of the fixed points of the calibration. Figure 9 shows the error propagation curves for the subrange 0 °C to the silver freezing point. The estimated NIST uncertainties (at the 1σ level) are ±1.0 m°C for the Sn, Zn and Al freezing points and ±2.0 m°C for the silver freezing point. The propagated error curve (for positive error only) for each fixed point used in the calibration is shown. The triple point of water error curve shown, based on an error of 0.1 m°C, is that incurred by the user, not an error in the NIST calibration. The thick line represents the total RSS error for the subrange, using the uncertainty associated with the NIST fixed points.

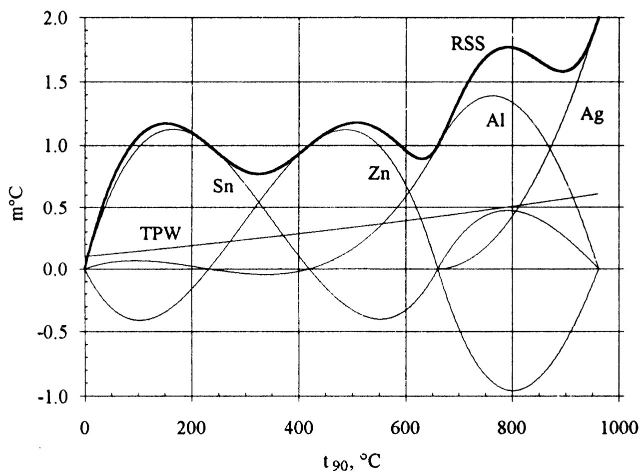

Figure 9. Propagation of error curve for the temperature subrange 0 °C to 961.78 °C. The propagated error for each fixed point is shown, with a triple point of water error being incurred by the user. The thick line represents the RSS error for that subrange based on the uncertainty of the NIST thermometric fixed points.

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[a] Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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